PATENT COOPERATION TREATY

PCT

INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY

(Chapter II of the Patent Cooperation Treaty)

(PCT Article 36 and Rule 70)

REC'D	0	4	MAY	2006

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Applicant's or agent's file reference	FOR FURTHER ACTION	See Form PCT/IPEA/416
International application No. PCT/GB2005/000311	International filing date (day/month/year, 28.01.2005	Priority date (day/month/year) 28.01.2004
International Patent Classification (IPC) of INV. C08J9/16 C08J9/28 C11D17	r national classification and IPC //00 C11D17/06	
Applicant UNILEVER PLC et al.		
, and the second of the second	reliminary examination report, established ansmitted to the applicant according to All of 7 sheets, including this cover sheet.	ed by this International Preliminary Examining Article 36.
3. This report is also accompanied		•
	to the International Bureau) a total of 15	
	tion, claims and/or drawings which have	book and the second of
Administrative Instruc	ctions).	been amended and are the basis of this report nority (see Rule 70.16 and Section 607 of the
Sheets which superse beyond the disclosure Supplemental Box.	ede earlier sheets, but which this Author e in the international application as filed,	ity considers contain an amendment that goes , as indicated in item 4 of Box No. I and the
b. (sent to the International is sequence listing and/or ta Relating to Sequence List	Bureau only) a total of (indicate type and bles related thereto, in electronic form o ting (see Section 802 of the Administration	d number of electronic carrier(s)) , containing a nly, as indicated in the Supplemental Box ve Instructions).
4. This report contains indications re	elating to the following items:	
Box No. I Basis of the rep	port	
☐ Box No. II Priority		
☐ Box No. III Non-establishm	nent of opinion with regard to novelty, inv	/entive step and industrial applicability
LI BOX NO. IV Lack of unity of	invention	a selection applicability
applicability, oil	ement under Article 35(2) with regard to a ations and explanations supporting such	novelty, inventive step or industrial statement
☐ Box No. VI Certain docume		
	in the international application	
⊠ Box No. VIII Certain observa	tions on the international application	
Date of submission of the demand	Date of completion	on of this report
20.07.2005	03.05.2006	
Name and mailing address of the internation oreliminary examining authority:	al Authorized officer	
European Patent Office		Supplied the s Patentam,
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INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY

International application No. PCT/GB2005/000311

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_	Bo	x No. I Basis of the repor	t			
ব.	Wit	With regard to the language, this report is based on				
	\boxtimes	★ The international application in the language in which it was filed				
		a translation of the international application into, which is the language of a translation furnished for the purposes of: international search (under Rules 12.3(a) and 23.1(b)) publication of the international application (under Rule 12.4(a)) international preliminary examination (under Rules 55.2(a) and/or 55.3(a))				
2. With regard to the elements * of the international application, this report is based on <i>(replacement shave been furnished to the receiving Office in response to an invitation under Article 14 are referred report as "originally filed" and are not annexed to this report):</i>						
	Des	scription, Pages				
	1, 4	, 5, 7-14, 16, 19-26	as originally filed			
	2, 2	ea, 3, 6, 15, 17, 18	filed with telefax on 27.07.2005			
	Clai	ims, Numbers				
	1-22	2	filed with telefax on 27.07.2005			
		a sequence listing and/or ar	ny related table(s) - see Supplemental Box Relating to Sequence Listing			
3.	 □ The amendments have resulted in the cancellation of: □ the description, pages □ the claims, Nos. □ the drawings, sheets/figs □ the sequence listing (specify): □ any table(s) related to sequence listing (specify): 					
4.	□ had Sup	This report has been estable not been made, since they has plemental Box (Rule 70.2(c)) the description, pages the claims, Nos. the drawings, sheets/figstone the sequence listing (specific any table(s) related to see	ecify):			
	*	If item 4 applies, so	ome or all of these sheets may be marked "superseded."			

INTERNATIONAL PRELIMINARY REPORT **ON PATENTABILITY**

International application No. PCT/GB2005/000311

Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial Box No. V applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)

Yes: Claims

No:

No:

Claims

1-23

Inventive step (IS)

Yes: Claims

Claims

1-23

Industrial applicability (IA)

Yes: Claims

1-23

No: Claims

2. Citations and explanations (Rule 70.7):

see separate sheet

Box No. VIII Certain observations on the international application

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:

see separate sheet

Reference is made to the following documents:

- D1: WO 03/091321 A (THE UNIVERSITY OF LIVERPOOL; COOPER, ANDREW, IAN; ZHANG, HAIFEI)
- D2: WO 99/00187 A (BIOPORE CORPORATION)
- D3: PATENT ABSTRACTS OF JAPAN vol. 013, no. 182 (C-591), 27 April 1989 (1989-04-27) -& JP 01 011141 A (NIPPI KORAAGEN KOGYO KK)
- **D4**: US-A-5 723 508 (HEALY ET AL)
- **D5**: WO 2004/011537 A (UNILEVER N.V; UNILEVER PLC; HINDUSTAN LEVER LIMITED; COOPER, ANDREW, I)
- D6: WO 2005/014704 A (THE UNIVERSITY OF LIVERPOOL; COOPER, ANDREW; BUTLER, RACHEL)

Re Item I

Basis of the report

1. Amendments

- 1.1 The amendments filed with the International Bureau under Art. 19(1) introduce subject-matter which extends beyond the content of the application as filed, contrary to Art. 19(2) PCT. The amendments concerned are the following:
- 1.2 The additional feature (c) "a hydrophobic material to be dispersed when the water-soluble polymeric material dissolves" has been introduced into amended independent claim 1. In contrast to this statement, page 6, lines 16-18 of the description as originally filed reads "...include within the lattice hydrophobic material to be dispersed when the bodies are dispersed in an aqueous medium". Nowhere in the description and claims as originally filed any support can be found for the added feature, that the polymer material dissolves.
- 1.3 Therefore, the amended claims 1-22 and amended description pages 2, 2a and 3 extend beyond the content of the application as filed.
- 1.4 Consequently, this report is based on the claims 1-23 as originally filed.

Re Item VIII

Certain observations on the international application

2. Clarity - Art. 6 PCT

- 2.1 Independent claim 1 as originally filed requires that the polymeric material should be water soluble. However, not all polymers claimed in dependent claim 3 fulfil this requirement. E.g. cellulose acetate and natural cellulose which also belong to the group of polysaccharides are water insoluble.
- **2.2** Expressions like "about" or similar terms like "a ambient temperature", "a temperature effective" and "a temperature effective" as used in claims 1, 6, 11 and 15 as originally filed are contrary to the requirements of PCT-Guidelines 5.38.
- 2.3 Some embodiments of the invention shown in the examples do not fall within the scope of the claims since they are silent concerning the value for the "intrusion volume" obtained after the freeze drying process. This inconsistency between the claims and the description leads to doubt concerning the matter for which protection is sought, thereby rendering the claims unclear, Art. 6 PCT.
- 2.4 The independent claim 11 as originally filed comprises the proviso "that said porous bodies are not spherical beads having an average bead diameter of 0.2-5 mm". In contrast, this feature is missing from the independent claim 1 of the present application.

Re Item V

Reasoned statement with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

3. Novelty

- 3.1 The independent claim 1 as originally filed claims
 - a) Water dispersible or water soluble porous bodies comprising
 - b) a three dimensional open-cell lattice containing less than 10 by weight of a water soluble polymeric material other than a surfactant, and
 - c) 5-95 % by weight of a surfactant,
 - d) said porous bodies having an intrusion volume as measured by mercury porosimetry of at least 3 ml*g⁻¹.
- 3.2 Document D1 discloses a) a porous crosslinked polymer material, which is therefore water dispersible, comprising b) a three dimensional open-cell lattice containing 1-80 % by weight of a water soluble polymeric material e.g. acrylic acid, acrylamide and c) 1-60 % by weight of a surfactant, e.g. SDS, d) having a pore volume of from 1-5 ml*g⁻¹. e) 50-100 % of the beads are substantially spherical. f) A process comprising a freeze drying step is not disclosed (abstract; pages 3-7).

- 3.3 Therefore, claims 1-10, 22 and 23 as originally filed do not fulfill the requirements of Art. 33(1) PCT since they are not novel over the prior art document **D1** in the sense of Art. 33(2) PCT.
- Document **D2** discloses **a)** a hydrophilic porous crosslinked and thus water dispersible polymeric material comprising a **b)** a three dimensional open-cell lattice containing a water soluble polymeric material e.g. acrylic acid, acrylamide and **c)** a surfactant. The amounts are within the range of the present application. **d)** The porous polymer particles show a bulk density within the range of from 0.001-1.0 g*ml⁻¹as also shown in the examples of the present application. **e)** At least approximately 10 % of the microbeads are substantially spherical or ellipsoidal or a combination thereof. **f)** A process comprising a freeze drying step is not disclosed (page 9, lines 30-33; page 11, lines 30-33; page 16, line 14-page 17, line 4; page 25, lines 19-28; Examples; claims 1, 6, 18, 22, 23).
- 3.5 Therefore, claims 1-10, 22 and 23 as originally filed do not fulfill the requirements of Art. 33(1) PCT since they are not novel over the prior art document **D2** in the sense of Art. 33(2) PCT.
- 3.6 Document **D3** discloses **a)** porous hydrophilic polymer articles comprising **b)** 0.05-50 % of a hydrophilic polymer like polyvinyl alcohol **c)** and 0.1-100 % of a surfactant. **d)e)** The porous articles are obtained by pouring the mixture into a tray, completely freezing it followed by a drying step (abstract).
- 3.7 Therefore, claims 1-23 as originally filed do not fulfill the requirements of Art. 33(1) PCT since they are not novel over the prior art document **D3** in the sense of Art. 33(2) PCT.
- 3.8 It is pointed out that documents **D5** and **D6** which have been cited in the ISR as a "P" and "E" document, respectively, might be relevant to the question of novelty in case that the application enters a regional and/or national phase.

4. Inventive Step

- **4.1** Beside the novelty objections raised above, the working-up of porous polymer materials by freeze drying is generally known in the art and can be found in documents **D3** and **D4** for example.
- 4.2 Therefore, it also seems that the whole set of claims as originally filed does not fulfil the requirements of Art. 33(1) PCT since it does not involve an inventive step in the sense of Art. 33(3) PCT.

INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY (SEPARATE SHEET)

International application No.

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- 5. Industrial Applicability
- 5.1 The subject matter of claims 1-23 as originally filed is industrial applicable.

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of a non-aqueous dispersed phase from a high internal phase emulsion. The beads are freeze-dried to remove the bulk of the aqueous phase. This leaves a 'skeletal' form of the emulsion behind. The beads dissolve rapidly in water and have the remarkable property that a water insoluble component dispersed in the emulsion prior to drying can also be dispersed in water on solution of the beads. Surfactant is present as an emulsifier.

10 Brief Description of the Invention

We have now determined that effectively polymer-free systems, i.e. which comprise a surfactant but little or no polymer can provide highly porous bodies which disperse rapidly on contact with water, even at low temperatures.

In accordance with a first aspect of the invention, there is provided water dispersible or water porous bodies comprising a three-dimensional <u>oil and water emulsion-templated</u> opencell lattice comprising:

- a) less that 10%wt of a water-soluble polymeric material other than a surfactant
- 25 b) 5-95%wt of a surfactant, and,
 - a hydrophobic material to be dispersed when the watersoluble polymer dissolves,
- said porous bodies having an intrusion volume as measured by mercury porosimetry of at least 3ml/g., and, with the

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proviso that they are not spherical beads having an average bead diameter of 0.2-5.0mm.

These porous bodies are particularly advantageous especially when
they contain a relatively high level of surfactant as this
promotes rapid dissolution of the surfactant and dispersion of

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the water insoluble material for which the surfactant is a carrier.

The present invention also provides water dispersible or water porous bodies comprising a three-dimensional <u>oil and</u>

<u>water emulsion-templated</u> open-cell lattice comprising:

- a) less that 10%wt of a water-soluble polymeric material other than a surfactant
- b) 5-95%wt of a surfactant, and,
 - a hydrophobic material to be dispersed when the watersoluble polymer dissolves,

said porous bodies having an intrusion volume as measured by mercury porosimetry of at least 3ml/g., and, with the proviso that they are not spherical beads having an average bead diameter of 0.2-5.0mm.

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The cooled emulsion retains its structure when the bulk of the phases are removed (preferably by freeze drying) leaving a solid, surfactant-containing lattice. This lattice retains its structure provided that the ambient temperature does not rise above its melting point. The lattice so produced is characterised by a large surface area, which greatly assists the solution of its components. This improved solution rate is particularly beneficial when the surfactant is being used for delicate cleaning tasks such as for cleaning delicate fabrics or where only cold water is available for use in the cleaning process.

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4 ml/g to about 25ml/g, more preferably from about 10 ml/g to about 20ml/g.

Intrusion volume provides a very good measure (in materials of this general type) of the total pore volume within the porous bodies of the present invention.

The porous bodies may be in the form of powders, beads or moulded bodies. Powders may be prepared by the disintegration of porous bodies in the form of beads or disintegration of bodies during other stages of the production process.

Porous bodies as carriers:

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The porous bodies of the present invention include within the lattice hydrophobic materials to be dispersed when the bodies are dispersed in an aqueous medium. Dispersion into an aqueous medium of such hydrophobic materials is much improved.

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The hydrophobic materials are incorporated into the lattice by dissolving them in the discontinuous oil phase of an oil-in-water emulsion from which the lattice is made.

The present invention also includes, in a further aspect, solutions or dispersions comprising surfactant and a hydrophobic material formed by exposing to an aqueous medium porous bodies according to the present invention, wherein said bodies comprise the hydrophobic material.

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Examples of water soluble polymeric materials include: -

- (a) natural polymers (for example naturally occurring gums such as guar gum or locust bean gum or a polysaccharide such as dextran;
- (b) cellulose derivatives for example xanthan gum, xyloglucan, cellulose acetate, methylcellulose, methylcellulose, hydroxyethylcellulose, hydroxyethylcellulose, hydroxy-propylcellulose, hydroxypropylmethylcellulose (HPMC), hydroxypropylmethylcellulose, ethylhydroxyethylcellulose, carboxy-methylcellulose and its salts (eg the sodium salt SCMC), or carboxymethylhydroxyethylcellulose and its salts (for example the sodium salt);
 - (c) homopolymers of any one of the monomers listed in Table 1 below;
- 20 d) copolymers prepared from two or more monomers listed in Table 1 below;
 - (e) mixtures thereof

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water dispersible nature of the resulting polymeric material.

Examples of suitable homopolymers include polyvinylalcohol,

polyacrylic acid, polymethacrylic acid, polyacrylamides
(such as poly-N-isopropylacrylamide), polymethacrylamide;
polyacrylamines, polymethylacrylamines, (such as
polydimethylaminoethyl-methacrylate and poly-Nmorpholinoethylmethacrylate, polyvinyl-pyrrolidone,

polyvinylimidazole, polyvinylpyridine, polyethylene-imine
and ethoxylated derivatives thereof.

Method of Preparation:

As noted above, one method suitable for preparing the porous bodies comprises the steps of: cooling a surfactant-containing oil-and-water emulsion to a temperature at which the continuous phase becomes solid, and subsequently removing the bulk of the continuous and dispersed phases.

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When the porous body is to be in the form of a powder the cooling of the liquid medium may be accomplished by spraying the liquid medium in atomised form into the fluid freezing medium.

When the porous body is to be in the form of beads the cooling of the liquid medium may be accomplished by dropping drops of the liquid medium into the fluid freezing medium.

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CLAIMS

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- 1) Water dispersible or water soluble porous bodies
 5 comprising a three dimensional oil and water emulsion templated open-cell lattice containing:
 - (a) less than 10% by weight of water-soluble polymeric material other than a surfactant, and
 - (b) 5 to 95% by weight of a surfactant, and,
 - (c) a hydrophobic material to be dispersed when the water-soluble polymeric material dissolves,

said porous bodies having an intrusion volume as measured by mercury porosimetry (as hereinafter described) of at least about 3 ml/g, with the proviso that they are not spherical beads having an average bead diameter of 0.2-5.0mm.

- 2) Porous bodies as claimed in claim 1 wherein the bodies are in the form of powders or moulded bodies
- Porous bodies as claimed in claim 1 or claim 2 wherein the polymeric material is a natural gum, a polysaccharide, a cellulose derivative or a homopolymer or copolymer comprising (co)monomers selected from:-vinyl alcohol,
- acrylic acid, methacrylic acid

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acrylamide, methacrylamide acrylamide methylpropane sulphonates aminoalkylacrylates 5 aminoalkylmethacrylates hydroxyethylacrylate hydroxyethylmethylacrylate vinyl pyrrolidone vinyl imidazole 10 vinyl amines vinyl pyridine ethyleneglycol ethylene oxide ethyleneimine styrenesulphonates 15 ethyleneglycolacrylates ethyleneglycol methacrylate

Porous bodies as claimed in claim 3 wherein the 4) cellulose derivative is selected from xanthan gum, 20 xyloglucan, cellulose acetate, methylcellulose, methyethylcellulose, hydroxyethyl-cellulose, hydroxyethylmethylcellulose, hydroxypropylcellulose, hydroxypropylmethylcellulose (HPMC), hydroxypropylbutylcellulose, 25 ethylhydroxyethylcellulose, carboxymethylcellulose and its salts, or carboxymethyl-hydroxyethylcellulose and its salts

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- 5) Porous bodies as claimed in any preceding claim wherein the surfactant is non-ionic, anionic, cationic, or zwitterionic
- 5 6) Porous bodies as claimed in any preceding claim wherein the surfactant is solid at ambient temperature
- Porous bodies as claimed in any preceding claim wherein 7) the surfactant is selected from ethoxylated 10 triglycerides; fatty alcohol ethoxylates; alkylphenol ethoxylates; fatty acid ethoxylates; fatty amide ethoxylates; fatty amine ethoxylates; sorbitan alkanoates; ethylated sorbitan alkanoates; alkyl ethoxylates; pluronics; alkyl polyglucosides; stearol 15 ethoxylates; alkyl polyglycosides; alkylether sulfates; alkylether carboxylates; alkylbenzene sulfonates; alkylether phosphates; dialkyl sulfosuccinates; alkyl sulfonates; soaps; alkyl sulfates; alkyl carboxylates; alkyl phosphates; paraffin sulfonates; secondary n-20 alkane sulfonates; alpha-olefin sulfonates; isethionate sulfonates; fatty amine salts; fatty diamine salts; quaternary ammonium compounds; phosphonium surfactants; sulfonium surfactants; sulfonxonium surfactants; Nalkyl derivatives of amino acids (such as glycine, betaine, aminopropionic acid); imidazoline surfactants; 25 amine oxides; amidobetaines; and mixtures thereof
- Porous bodies as claimed in any preceding claim wherein the porous polymeric bodies have water soluble materials incorporated into the polymeric lattice

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- Water soluble porous polymeric bodies as claimed in 9) claim 8 wherein the water soluble material is selected from water soluble vitamins; water soluble fluorescers; activated aluminium chlorohydrate; transition metal complexes used as bleaching catalysts; water soluble polymers; diethylenetriaminepentaacetic acid (DTPA); primary and secondary alcohol sulphates containing greater than C8 chain length or mixtures thereof
- Water soluble porous polymeric bodies as claimed in 10 10) claim 1 wherein the water insoluble material is selected from antimicrobial agents; antidandruff agent; skin lightening agents; fluorescing agents; antifoams; hair conditioning agents; fabric conditioning agents; skin conditioning agents; dyes; UV protecting agents; 15 bleach or bleach precursors; antioxidants; insecticides; pesticides; herbicides; perfumes or precursors thereto; flavourings or precursors thereto; pharmaceutically active materials; hydrophobic polymeric materials and mixtures thereof. 20
 - 11) A method for preparing water dispersible or water soluble porous bodies comprising a three dimensional oil-and-water emulsion-templated open-cell lattice containing
 - less than 10% by weight of a water soluble (a) polymeric material and

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- (b) 5 to 90% by weight of a surfactant, and a hydrophobic material to be dispersed when the water-soluble polymer dissolves
- said porous bodies having an intrusion volume as measured by mercury porosimetry (as hereinafter described) of at least about 3 ml/g with the proviso that said porous bodies are not spherical beads having an average bead diameter of 0.2 to 5mm

comprising the steps of:

- a) providing an oil-in-water emulsion comprising the polymeric material, the hydrophobic material and the surfactant in a liquid medium
 - b) providing a fluid freezing medium at a temperature effective for rapidly freezing the liquid medium;
- c) cooling the liquid medium with the fluid freezing medium at a temperature below the freezing point of the liquid medium for a period effective to rapidly freeze the liquid medium; and
- d) freeze-drying the frozen liquid medium to form the porous bodies by removal of the liquid medium by sublimation.
- 12) A method as claimed in claim 11 wherein the cooling of the liquid medium is accomplished by spraying an atomised emulsion into the fluid freezing medium; by

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dropping drops of the emulsion into the fluid freezing medium or by pouring the emulsion into a mould and cooling the emulsion in the mould.

- 5 13) A method as claimed in claim 11 or 12 wherein the polymeric material is a natural gum, a polysaccharide, a cellulose derivative or a homopolymer or copolymer comprising (co)monomers selected from:-
- vinyl alcohol,
 acrylic acid,
 methacrylic acid
 acrylamide,
 methacrylamide
- acrylamide methylpropane sulphonates
 aminoalkylacrylates
 aminoalkylmethacrylates
 hydroxyethylacrylate
 hydroxyethylmethylacrylate
- vinyl pyrrolidone vinyl imidazole vinyl amines vinyl pyridine ethyleneglycol
- ethylene oxide
 ethyleneimine
 styrenesulphonates
 ethyleneglycolacrylates
 ethyleneglycol methacrylate

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- 14) A method as claimed in any one of claims 11 to 13 wherein the surfactant is non-ionic, anionic, cationic, or zwitterionic
- 5 15) A method as claimed in any one of claims 11 to 14 wherein the surfactant is solid at ambient temperature
 - 16) A method as claimed in any one of claims 11 to 15wherein the surfactant has an HLB value of 8 to 18

- 17) A method as claimed in any one of claims 11 to 16
 wherein the surfactant is selected from ethoxylated
 triglycerides; fatty alcohol ethoxylates; alkylphenol
 ethoxylates; fatty acid ethoxylates; fatty amide
 ethoxylates; fatty amine ethoxylates; sorbitan
 alkanoates; ethylated sorbitan alkanoates; alkyl
 ethoxylates; pluronics; alkyl polyglucosides; stearol
 ethoxylates; alkyl polyglycosides; alkylether sulfates;
 alkylether carboxylates; alkylbenzene sulfonates;
 alkylether phosphates; dialkyl sulfosuccinates; alkyl
 sulfonates; soaps; alkyl sulfates; alkyl carboxylates;
 alkyl phosphates; paraffin sulfonates; secondary nalkane sulfonates; alpha-olefin sulfonates; isothionate
- alkane sulfonates; alpha-olefin sulfonates; isethionate sulfonates; fatty amine salts; fatty diamine salts; quaternary ammonium compounds; phosphonium surfactants; sulfonium surfactants; sulfonium surfactants; N-alkyl derivatives of amino acids (such as glycine, betaine, aminopropionic acid); imidazoline surfactants; amine oxides; amidobetaines; and mixtures thereof

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- 18) A method as claimed in claim 11 wherein the discontinuous phase of the emulsion comprises 10 to 95% by volume of the emulsion
- 5 19) A method as claimed in claim 11 wherein the discontinuous phase of the emulsion comprises 20 to 60% by volume of the emulsion
- 20) A method as claimed in claim 11 wherein the

 discontinuous phase of the emulsion is selected from

 alkanes; cyclic hydrocarbons; halogenated alkanes;

 esters; ketones; ethers; volatile cyclic silicones and

 mixtures thereof
- 15 21) Solutions or dispersions comprising water soluble polymeric materials and surfactant formed by exposing the porous bodies of any one of claims 1 to 10 to an aqueous medium.
- 20 · 22) Solutions or dispersions comprising water soluble polymeric materials, surfactant and a hydrophobic material formed by exposing the porous bodies of claim 1 having the hydrophobic material contained therein to an aqueous medium.